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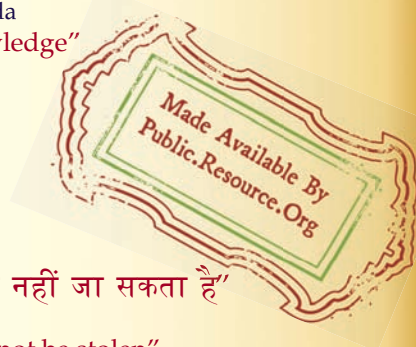
IS 12107-6 (1987): Methods of chemical analysis of alumino-silicate refractory materials, Part 6: Determination of iron [MTD 13: Ores and Raw Materials]



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“Knowledge is such a treasure which cannot be stolen”

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(Reaffirmed 2001)

Indian Standard

METHODS OF CHEMICAL ANALYSIS OF ALUMINO SILICATE
REFRACTORY MATERIALS

PART 6 DETERMINATION OF IRON

1. Scope — This standard (Part 6) covers a method for determination of iron (as Fe_2O_3) in aluminosilicate refractory materials.

2. Determination of Iron by Spectrophotometric Method

2.1 Outline of the Method — Iron (III) in an aliquot from the main solution is reduced to iron (II) with hydroxylamine-hydrochloride and treated with *o*-phenanthroline. The orange red complex produced is measured at 510 nm in a spectrophotometer. The amount of iron (as Fe_2O_3) is evaluated from the calibration curve obtained under similar conditions.

2.2 Reagents

2.2.1 *Paranitrophenol indicator solution* — 1 percent (*m/v*).

2.2.2 *Tartaric acid solution* — 10 percent (*m/v*).

2.2.3 *Dilute ammonia solution* — 1:3 (*v/v*).

2.2.4 *Dilute hydrochloric acid* — 1:1 (*v/v*).

2.2.5 *Hydroxylamine hydrochloride* — 10 percent (*m/v*).

2.2.6 *o-phenanthroline* — 0.1 percent (*m/v*) in ethanol.

2.2.7 *Standard iron solution* — Dissolve 0.4911 g of ferrous ammonium sulphate in water and add 10 ml of dilute sulphuric acid (1 : 1) and 5 ml of hydrogen peroxide. Boil for 15 minutes to decompose the excess of hydrogen peroxide. Cool and dilute to 1 litre in a volumetric flask (1 ml = 0.1 mg Fe_2O_3).

2.3 Procedure

2.3.1 Transfer 5 ml aliquot of the main solution [see 2.3.3 of IS : 12107 (Part 2)-1987 Methods of chemical analysis of aluminosilicate refractory materials : Part 2 Determination of silica] into 100-ml volumetric flask. Add 1-2 drops of paranitrophenol indicator and 10 ml of tartaric acid solution. Neutralize the solution by dropwise addition of ammonia solution to the yellow colour. Acidify with a few drops of dilute hydrochloric acid to pH 4.0-4.5. Add 2 ml of hydroxylamine hydrochloride and 10 ml of *o*-phenanthroline. Cool the solution in the flask and make up the volume. Allow to stand for 1 hour at room temperature.

2.3.2 Measure the absorbance at 510 nm against a reagent blank prepared in the same way without the sample aliquot.

2.3.3 *Calibration curve* — Draw a calibration curve by taking 0, 1, 2, 3, 4 and 5 ml of standard iron solution (1 ml = 0.1 mg Fe_2O_3) in five 100-ml volumetric flasks. Proceed in accordance with 2.3.1 and measure the absorbance of each solution at 510 nm against the reagent blank. Plot the absorbance values against mg of Fe_2O_3 /100 ml of the solution.

Adopted 24 August, 1987

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Gr 1

2.4 Calculation — Convert the photometric readings of the sample to mg of Fe_2O_3 by means of the calibration curve and calculate Fe_2O_3 content as follows:

$$\text{Iron (as } \text{Fe}_2\text{O}_3 \text{), percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mass in mg of Fe_2O_3 found in 5 ml aliquot of the final solution, and

B = mass in g of the sample represented by the aliquot taken.

EXPLANATORY NOTE

Alumino silicate refractory materials contain alumina (Al_2O_3) and silica (SiO_2) in varying portions made synthetically by heating aluminium trifluoride at 1 000-1 200°C with silica and water vapour.

It is used in kilns, laddles and furnace that operate at higher temperature or under conditions for which fire clay refractories are not suitable.

This Indian Standard has been prepared in different parts to cover the chemical analysis of various constituents in alumino silicate refractory materials. The other parts of the standard are:

Part 1 Determination of loss on ignition

Part 2 Determination of silica

Part 3 Determination of aluminium

Part 4 Determination of phosphorus

Part 5 Determination of titanium

Part 7 Determination of manganese

Part 8 Determination of calcium and magnesium

Part 9 Determination of sodium and potassium